

⁵⁰Ti for a High-Intensity Heavy-Ion Beam

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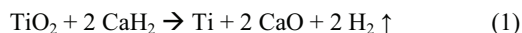
The semi-magic ⁵⁰Ti has a closed neutron shell with N = 28 and is the most neutron rich of the stable Ti-isotopes. This makes the isotope especially interesting as a beam projectile for heavy-element synthesis. For the production of a high intensity ion beam over long time periods, the metallic form of the enriched isotope is preferable for both, ECR and PIG ion sources.

Highly enriched ⁵⁰Ti is delivered either as the dioxide or as the tetrachloride. The task was to establish a process to reduce the compound to the metal with a high efficiency and a high chemical purity.

Process

For the reduction process we preferred the solid TiO₂ to the liquid TiCl₄ since handling gaseous TiCl₄ at higher temperatures needed for the reduction process involves a higher risk of losing material. For the TiO₂ there are in principle two common routes for reducing it to the metal either with metallic Ca or with CaH₂ as reducing agent [1]. Since for the affectivity of the reduction the purity of the Ca is crucial we decided to use the CaH₂ in the process. The Ca vapours are generated within the mixture via thermal decomposition of the hydride and can react immediately with the adjacent TiO₂.

The process chosen [2] is described by the following chemical equation:



For the reduction we prepared a mixture of the two components in an agate mortar with a 40% excess of CaH₂ to the stoichiometric composition described by the reaction equation (1) and pressed to tablets with a hydraulic press. The tablets are heated in a molybdenum boat inside a stainless steel tube at 950°C for an hour in a constant flow of dry argon. After the reduction the tablets now containing Ti-metal, CaO and rest of Ca are cooled down to room temperature, dissolved in diluted acetic acid; then the precipitate is washed and dried, obtaining a fine powder of metallic Ti.

Analysis

At first the setup and the process were tested with different batches of natural TiO₂ where a yield of 95 % was achieved. For the application in the ion sources ten different batches of ⁵⁰TiO₂ from three different producers were reduced. The starting material obviously differed in colour, in grain size and in softness leading to quite different

behaviour by pressing tablets for the reduction and by melting metallic Ti, as well as a significant variation in yield during reduction.

To get an understanding of the impurities in the starting material and their behaviour during the reduction process, energy-dispersive x-ray analysis (EDX) of all batches before and after the reduction was applied. In the natural material no impurities were detectable before the reduction, after the reduction a Ca-content of ~ 0.3 – 0.6 % was observed. Several batches of the enriched ⁵⁰TiO₂ showed no impurities before and only the expected amount of calcium after the reduction. But a number of batches had impurities of Cl, Si or Sn or a combination of those. All impurities were in the range of 1.5 % up to 5 %. The Cl vanished completely after the reduction, whereas the Si and the Sn stayed in the same amount.

The different impurities caused different behaviour in the further processing especially by melting tablet of Ti powder to beads.

Results

With the new setup we are able to convert between 0.5 g up to several grams of highly enriched ⁵⁰Ti in one run with yields between 90 % and 98 %. Different impurities in the starting material influence the melting behaviour and therefore the final yield significantly. We obtain enriched metallic titanium for application in the ion source or as a starting material for target production. The reduced material was used at UNILAC for production of ⁵⁰Ti-beam with intensities of about 1 particle/μA at a target for experiments which lasted several months in 2011 and 2012 [3,4,5].

References

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